FOURTH QUARTERLY TECHNICAL PROGRESS REPORT

ALKALI METAL RESISTANT ELECTRICAL DEVICES

USAF CONTRACT AF33(615)-3528
BUDGET NO. (BPSN) 6(638128 62405214)

JULY 15, 1967
WAED 67.29E

Westinghouse Electric Corporation
Aerospace Electrical Division
Lima, Ohio

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(26 March 1966 - 25 June 1967)

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FOREWORD

This fourth quarterly report is submitted by the Aerospace Electrical Division, Westinghouse Electric Corporation, Lima, Ohio, on Air Force Contract AF33(615)-3528, Budget No. (BPSN) 6(638128 62405214) Alkali Metal Resistant Electrical Devices. The contract is administered by the Air Force Aero Propulsion Laboratory, Research and Technology Division, Wright-Patterson Air Force Base, Dayton, Ohio. Mr. Lester Schott is Project Engineer for APIE on this contract.

The work described in this report was done by personnel in the Materials Development and Research and Development Groups of Westinghouse Aerospace Electrical Division, Lima, Ohio. The engineers and their areas of responsibility, in which they contibuted are as follows: A. J. Krause - Test Engineering and Mechanical Design, R. E. McVay - Metallurgical Studies, N. K. Harpster - Circuit Design, and R. E. Stapleton - Ceramic Technology. W. H. Snavely was the Project Engineer and R. M. Frost was the Program Manager.

ABSTRACT

This report covers the fourth quarter from 26 March 1967 to 25 June 1967 on Air Force Contract AF33 (615)-3528, Alkali Metal Resistant Electrical Devices.

Mechanical test data on metallic and ceramic specimens after 1000 hours in 600°C potassium are given. A nickel clad silver conductor was tested for 917 hours in potassium vapor with little increase in resistivity. Helium leak tight brazes were obtained on a Rowland ring core box ceramic part. Density and grain size measurements are given for WAED hot pressed ceramic materials. Alumina-yttria and alumina from aluminum oxychloride were applied to rhodium conductors.

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SECTION I

INTRODUCTION

This report covers the fourth quarter from 26 March 1967 25 June 1967 on Air Force Contract AF33(615)-3528, Alkali Metal Resistant Electrical Devices.

This program involves the study and evaluation of electrical device material for 600°C, 5000-hour, potassium vapor operation. Conductors, insulators, and magnetic materials capable of being used in potassium vapor exposed electrical circuits will be exposed to 600°C potassium vapor for times up to 5000 hours. Positive identification of degradation over this time period will be established with the goal of providing materials compatible with environmental test conditions. Processes and techniques necessary to insure mechanical and electrical compatibility and integrity of metal-to-metal and ceramic-to-metal interfaces will be investigated. The goal is to provide processing techniques for potassium vapor resistant electrical device fabrication. Transformers rated at 5 KVA will be fabricated and results of electrical tests in ionized and non-ionized 600°C potassium vapor will be evaluated.

SECTION II

SUMMARY OF WORK PERFORMED AND MAJOR RESULTS

This section provides a summary of the work performed and the major results obtained on the program in three general areas.

A. EVALUATION OF ELECTRICAL DEVICE MATERIALS IN 600°C POTASSIUM VAPOR

1. Long Term Potassium Vapor Exposure Tests

Loadings for long term potassium vapor exposure tests were completed and all tests begun. The 1000-hour test was completed and specimen evaluation was started. Test data and preliminary specimen evaluation are given.

2. Conductor Continuous Performance Tests

The two-terminal test container was given additional tests. The four-terminal test container was run for a total of 917 hours at a temperature of 600°C or higher before the test was terminated.

3. Rowland Ring Core Box Fabrication

Additional Rowland ring core box designs were prepared and braze attempts made on several of them. Helium leak tight brazing was accomplished on the terminal seal type core box.

B. PROCESSES AND TECHNIQUES FOR POTASSIUM VAPOR RESISTANT ELECTRICAL DEVICE FABRICATION

1. Materials for Short-Term Potassium Vapor Exposure Tests

Specimens of ceramics and ceramics on metals were prepared for tests. Some of these were loaded in potassium containing test capsules.

2. Ceramic/Metal Interface Development

Attempts were made to bond alumina to Hiperco 27 alloy substrates by reaction with titanium. These were unsuccessful; probably due to the binder used in applying titanium hydride. Alumina was applied to rhodium by the decomposition of aluminum oxychloride giving a non adhearing sintered coating. Pre-fused alumina-yttria powder was prepared and applied to rhodium wire giving a non-continuous coating of adhering fused droplets.

3. Properties of Hot-Pressed Materials

Density and grain size measurements were made on ceramic materials which were hot pressed at Westinghouse. Densities greater than 95% of theoretical were measured on these materials. Grain sizes of 25 microns or less were measured on all materials with the exception of alumina-yttria which appeared to be a two phase structure.

C. FABRICATION AND TESTING OF FIVE-KVA TRANSFORMERS

1. Transformer Magnetic Material

Work was done toward establishing the parameters, techniques, and equipment necessary to obtain a suitable interlaminar insulation by plasma arc spraying high purity alumina in an inert atmosphere chamber.

2. Transformer Electrical Conductors

A winding fixture was prepared and used in winding one layer of transformer conductors. These conductors were vacuum annealed to reduce springback.

3. Sapphire Window Seals

Tapered sapphire windows were brazed to columbium-1% zirconium tube parts using two different furnaces and various braze time-timperature cycles. Good wetting was accomplished, but leak tight joints with unbroken sapphire windows were not obtained.

SECTION III

EXPERIMENTAL WORK

This section describes the experimental work conducted on the program in three general areas.

A. EVALUATION OF ELECTRICAL DEVICE MATERIALS IN 600°C POTASSIUM VAPOR

1. General Description

Various magnetic materials, electrical conductors, and insulation materials are being exposed to 600°C potassium vapor for various times through 5000 hours and will be evaluated to determine degradation. The potassium used for these tests contains less than 25 ppm of oxygen. Most materials will be tested in quadruplicate:

a) As-prepared unexposed material.

b) After 1000 hours in 600°C potassium vapor.

c) After 2000 hours in 600°C potassium vapor.

d) After 3000 hours in 600°C potassium vapor.

e) After 5000 hours in 600°C potassium vapor.

After potassium vapor exposure, specimens will be treated as follows and evaluated by comparison with unexposed specimens:

a) Electrical conductors will be subjected to tensile testing, weight change determinations and visual and metallographic examinations.

b) Magnetic materials will be subjected to weight change determinations, tensile testing, and visual and metallographic examinations.

c) Ceramic Bar materials will be subjected to modulus of rupture tests, weight change determinations, and microscopic examinations.

d) Sapphire mat materials will be subjected to microscopic examinations.

e) Potassium from certain of these tests will be analyzed to determine oxygen content and impurity pick-up from the specimens.

A magnetic material will be subjected to magnetic tests in a potassium vapor atmosphere Rowland ring core box.

Electrical conductors will be given continuous electrical performance tests in a potassium vapor atmosphere.

Progress in the area of evaluating electrical device materials in potassium vapor is described below.

2. Long Term Potassium Vapor Exposure Tests

Loadings for long term potassium vapor tests were completed. The 1000-hour test was completed, capsules were unloaded, and specimen evaluation was started.

a. Loading For The 2000-Hour Test

Specimens for the 2000-hour test were loaded as those for the 1000, 3000, and 5000-hour tests (see Fourth Technical Progress Report[1]*) in either columbium-1% zirconium or stainless steel capsules containing high purity potassium. Test materials and capsule materials for these and other test durations are shown in Table I. Loading the 2000-hour test capsules completed the loadings for the long term potassium vapor exposure tests.

b. Unloading The 1000-Hour Test

The 1000-hour test container was removed from a 600°C circulating furnace after 1005 hours at temperature. The test container was free convection cooled to room temperature in the vertical position (the same position as during the test) in an attempt to condense and solidify the potassium in the liquid region of the capsule.** After cooling, the outer test container was opened and the test capsules examined for evidence of potassium. There was no evidence of potassium on the outside of the test capsules.

Potassium containing capsules from the 1000-hour test (both hot standards and specimen containing) and the potassium cold standards were taken to Technical Services Laboratory, Westinghouse Atomic Power Division, for unloading and potassium analysis. Cold standards are capsules of columbium-1% zirconium and 347 stainless steel loaded and sealed as the test capsules, but

* Superscripts refer to references in Section V.

** See figure 4 page 13 of First Quarterly Technical Progress Report[2].

TABLE I. LONG-TERM POTASSIUM VAPOR EXPOSURE TEST AND CONTROL SPECIMENS

Specimen*	Capsule Material	Capsule Atmosphere	600°C Test Duration (Hours) 1000	2000	3000	5000
Fused Strontium Zirconate	347 Stainless Steel	Potassium	C	0	0	0
Calcined Strontium Zirconate	347 Stainless Steel	Potassium	C		0	0
Alite A-610	347 Stainless Steel	Potassium	C	0	0	0
Beryllia	347 Stainless Steel	Potassium	C	0	0	0
Coors AD999	347 Stainless Steel	Potassium	C	0	0	0
Felted Sapphire Mat	347 Stainless Steel	Potassium	C	0	0	0
Alumina-Yttria Eutectic	347 Stainless Steel	Potassium			0	0
Yttria	347 Stainless Steel	Potassium	C			0
Boron Nitride Felt	Cb-1% Zr	Potassium				0
Hiperco 27 Alloy	Cb-1% Zr Stainless Steel	Potassium Vacuum	C C	0 0	0 0	0 0
Cubex	Cb-1% Zr Stainless Steel	Potassium Vacuum	C C	0 0	0 0	0 0
Nickel Clad Silver	Cb-1% Zr Stainless Steel	Potassium Vacuum	C	0 0	0 0	0 0
Inconel Clad Silver	Cb-1% Zr Stainless Steel	Potassium Vacuum	C C	0 0	0 0	0 0
Rhodium	Cb-1% Zr Stainless Steel	Potassium Vacuum	C C	0 0	0 0	0 0
Iridium	Cb-1% Zr Stainless Steel	Potassium Vacuum	C C	0 0	0 0	0 0
Hot Potassium Standard	Cb-1% Zr Stainless Steel	Potassium Potassium	C C	0 0	0 0	0 0

*A more complete description of metallic specimens is given in Table III.

0 = On Test
C = Test Completed

stored at room temperature and are the references for potassium impurity change due to capsule thermal exposure. Potassium samples from specimen containing capsules are being analyzed for metallic impurities by mass spectrograph techniques so that any impurity pick-up from the specimens can be determined. Potassium cold and hot standards are being analyzed for both metallic impurities and oxygen contents.

Exteriors of capsules were cleaned with acetone as the first step in the potassium unloading sequence. Cleaned capsules were then placed in an inert atmosphere glove box vestibule where evacuation and back filling were performed prior to passing the capsules into the glove box. Capsules were handled in the glove box in a high purity helium atmosphere. Oxygen level in the glove box atmosphere was maintained between 2 and 3 ppm and the moisture content was maintained at 0.15 ppm during the unloading of the specimen containing capsules. Specimen containing capsules were opened by circumferential cuts made with a tubing cutter.

Specimens were removed from their capsules by heating the potassium and pouring the specimens and potassium into glass vessels. Capsule parts were also placed in the glass vessels open end up. All vessels were sealed.

The following observations were made during the opening of specimen containing capsules:

1) All specimens had a continuous coating of potassium on their surfaces.

2) All potassium had a high luster.

3) The majority of the potassium was found in the vapor or specimen region of each capsule.

Potassium was prepared for metallic impurity analysis by removing the sealed glass vessels from the glove box and neutralizing the potassium with methyl alcohol under flowing argon. Specimens and tube interiors were rinsed with methyl alcohol to remove any insoluble impurities. All impurities and solutions were placed in suitable plastic evaporating beakers for subsequent evaluation.

Hot and cold potassium standard capsules were prepared by cleaning as were the specimen containing capsules and were handled singly in the helium atmosphere glove

box. Two potassium samples were removed from each capsule for oxygen analysis. Samples were removed by cutting the tube through the potassium in two places. Each oxygen sample was reacted with mercury and then processed by standard methods for oxygen determination by titration and by flame. The remaining potassium in each capsule was processed for metallic impurity analysis as described above.

c. Results and Comparisons of Mechanical Test Data

Mechanical tests were performed on most materials from the 1000-hour potassium vapor exposure tests. Tensile strength, yield strength, and elongation were determined on metallic specimens. Modulus-of-rupture values were determined for all the ceramic bars except one chemically pure strontium zirconate bar, which was found to be broken in unloading, and all the Alite A-610 bars, which broke before they could be weighed. No mechanical tests were performed on the sapphire mat.

Table II gives the modulus-of-rupture values and weight changes of the potassium exposed modulus-of-rupture bars as well as the modulus-of-rupture values of the unexposed standards. The Coors AD999 decreased nearly one-fifth in modulus of rupture. Pre-fused strontium zirconate and chemically pure strontium zirconate decreased one-third in modulus of rupture. Beryllia and yttria showed very little change in modulus of rupture. Alite A-610 had essentially no strength with the bars crumbling during weighing prior to a possible modulus-of-rupture test. Previous potassium vapor exposure of the commercial Alite A-610 ceramic body indicated it would withstand corrosion by the 600°C potassium vapor. As these specimens were severly attacked by the potassium vapor, analysis of the ceramic for silica will be done to determine if this batch of A-610 has a high silica content.

The sapphire mat was observed to have become darkened with a few black specs in it.

Results of mechanical tests on metallic specimens are given in tables III and IV. Tensile strengths, yield strengths, and elongations are given for the unexposed standard specimens, 1000-hour, 600°C vacuum exposed specimens, and 1000-hour, 600°C potassium vapor exposed specimens.

The exceptionally high strength of the as-received iridium wire is characterized by the fibrous texture

TABLE II. MODULUS-OF-RUPTURE VALUES OF UNEXPOSED AND 1000-HOUR POTASSIUM VAPOR EXPOSED CERAMICS

Material	Sample No.	Unexposed Modulus of Rupture				After 1000-Hour 600°C Potassium Exposure							Observations on Specimens as Neutralized From Potassium	Change in Average Modulus of Rupture (%)
						Modulus of Rupture				Specimen Weight				
		Modulus of Rupture (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Values (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Total Initial (gram)	Total Change (gram)	Total Change (%)		
BeO	1	20,625				[illegible]								
	2	19,800				144								
	3	25,000	22,556.5	2363	10.5	20,032	21,610	1241	5.7	3.6330	increase 0.0002	increase 0.01	White color like granules of sugar or salt.	Decreased 3.7
	4	24,800				22,560								
Alite A-610	1	32,600				-- *	--	--	--					
	2	38,000				--	--	--	--		decrease	decrease	One bar broken in handling. All surfaces crased. Some fractures showing. Dark gray color	Decreased to zero
	3	36,400	35,850	2164	6.05	--	-	--	--	2.5288	0.0227	0.897		
	4	36,400				--	--	--	--					
Coors AD999	1	49,500				50,794								
	2	67,000				54,071					increase	increase	Gray color.	Decreased 18.7
	3	70,000	63,125	8003	12.7	48,961	51,275	1897	3.6	2.3363	0.0004	0.02		
	4	66,000				--								
Y_2O_3	1	21,800	--	--	--	22,134								
	2	--	--	--	--	17,971					decrease	decrease	Slightly translucent. Light orange or pink color. Black color on one or both ends.	No apparent change
	3	--	--	--	--	23,982	22,188	2134	9.6	3.2620	0.0121	0.37		
	4	--	--	--	--	24,650								
Pre-fused $SrZrO_3$	1	32,700				25,612								
	2	27,600				23,096					increase	increase	Dark gray color. Mottled.	Decreased 29.4
	3	35,500	32,900	3290	10.0	21,624	23,204	1204	5.1	3.4401	0.0017	0.05		
	4	35,800				22,481								
Chemically Pure $SrZrO_3$	1	16,000				12,524								
	2	18,000				8,531							One end of one bar broke into small particles which were found in the potassium region of the capsule during unloading.	Decreased 33.2
	3	15,250	17,412	1996	11.4	13,823	11,626	2063	17.7	3.6330				
	4	20,400				--								

* These specimens crumbled during handling prior to weighing.

TABLE III. YIELD STRENGTH AND ELONGATIONS OF STANDARD AND 1000-HOUR EXPOSED METALLIC SPECIMENS

Material	No.	Unexposed Standards					1000-hr. 600°C Vacuum					1079-hr. 600°C Potassium Vapor				
		Yield Strength* (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Elongation (% in 2 in.)	Yield Strength* (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Elongation (% in 2 in.)	Yield Strength* (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Elongation (% in 2 in.)
0.011-inch thick Cubex Strip, Westinghouse (0.25" x 8")	1	41,851	41,971	681.25	1.6	22.5	41,851	39,634	1022.25	2.5	3	38,418	40,502	2469.75	6.4	14
	2	43,333				13.5	38,433				12.5	40,404				12.5
	3	41,218				15.0	39,403				10.0	32,682				25.0
	4	41,681				17.5	39,048				18.0	38,503				7.0
0.008-inch thick Hiperco 27 Alloy Strip, Westinghouse (0.25" x 8")	1	33,183	34,074	1712.5	5.0	21.0	34,756	33,113	2,340.25	7.2	3	35,960	35,922	612.5	1.7	**
	2	37,500				22.0	33,203				3	34,975				1
	3	32,433				22.5	27,317				3	37,109				6
	4	33,183				18.7	32,948				6	35,644				4
0.030-inch diameter Rhodium Wire, Sigmund Cohn (8" length)	1	22,857	20,535	1439.75	6.9	13.2	27,142	26,786	1069.75	3.9	12.5	25,714	23,571	3214.25	13.6	10
	2	19,642				13.0	25,714				13.5	17,142				12.5
	3	21,071				14.0	26,571				15.0	24,285				8.5
	4	18,571				13.5	25,714				11.0	27,142				14.0
0.030-inch diameter Iridium Wire, Englehard (8" length)	1	206,571	211,785	4642.75	2.1	10.0	238,571	246,428	3925.75	1.5	9.0	242,857	248,928	3214	1.8	8.5
	2	218,571				10.0	250,000				12.5	250,000				7.5
	3	205,714				15.0	250,000				8.5	254,285				15.0
	4	214,285				9.0	247,142				7.5	248,571				11.0
0.101-inch diameter Inconel Clad Silver Wire, 20% cladding, Sylvania (8" length)	1	30,375	31,281	484.25	1.5	18.5	28,252	28,469	217.0	0.7	24.0	27,633	28,100	480.5	1.7	25.0
	2	31,250				22.0	28,252				19.0	27,681				22.5
	3	32,250				21.5	28,672				25.0	28,004				20.0
	4	31,250				22.0	26,500				24.5	29,120				24.0
Rectangular Nickel Clad Silver Wire, 20% Cladding, Sylvania (0.125" x 0.060" x 8")	1	[illegible]37	34,730	613.0	1.7	23.0	16,336	18,255	2676.25	14.6						
	2	36,098				18.5	13,366									
	3	34,807				15.0	14,356									
	4	35,588				15.0	19,801									
	5						20,495									
	6						20,792									
	7						20,594									
	8						20,297									

* Yield Strength at 0.2% Offset.

** A fragment of this specimen was lost.

TABLE IV. TENSILE STRENGTH OF STANDARD AND 1000-HOUR EXPOSED METALLIC SPECIMENS

Material	No.	Unexposed Standards				1000-hr. 600°C Vacuum				1000-hr. 600°C Potassium Vapor			
		Tensile Strength (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Tensile Strength (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)	Tensile Strength (psi)	Average (psi)	Standard Deviation (psi)	Coefficient of Variation (%)
Cubex	1	43,333				48,227				45,807			
	2	48,516				47,584				46,897			
	3	47,850	47,055	1860.75	3.9	46,800	47,634	441.5	0.9	47,957	47,450	1097.75	2.3
	4	48,518				47,923				48,150			
Hiperco	1	71,748				60,327				65,024			
	2	72,632				58,105				54,213			
	3	72,072	72,249	364.25	0.5	56,585	58,235	2714.75	4.6	66,496	65,024	4094.25	6.2
	4	72,645				64,922				63,964			
Rhodium	1	71,428				67,142				74,285			
	2	66,428				71,428				77,142			
	3	62,142	66,312	2366.5	3.5	71,428	69,285	2143	3.0	75,000	75,178	982	1.3
	4	66,248				67,142				74,285			
Iridium	1	574,285				295,714				294,285			
	2	564,285				297,142				281,428			
	3	574,285	576,428	8214.5	1.4	291,428	299,642	6816.25	2.2	302,857	291,071	7500	2.5
	4	592,857				314,285				285,714			
Inconel Clad Silver	1	47,500				46,840				46,220			
	2	48,000				46,468				47,087			
	3	47,500	47,750	250	0.5	47,459	46,964	307	0.6	45,105	46,375	712.25	1.5
	4	48,000				47,087				47,087			
Nichel Clad Silver	1	37,549				30,990							
	2	38,235				31,188							
	3	37,647	38,162	564	1.4	30,990							
	4	39,217				30,990							
	5					30,990	31,114	123.75	0.3				
	6					31,188							
	7					31,188							
	8					31,386							

* A more complete description of specimens is given in Table III.

shown in figure 1. Figures 2 and 3 show microstructure at 384X. The as-received material was evidently not annealed as was specified. The thermal exposure and potassium exposure samples both showed about 50% decrease in tensile strength and about a 17% increase in yield strength as compared to the unexposed standards. Complete analysis of metallic specimens has not been completed.

Table V gives the weights of metallic specimens both before and after 1000-hour, 600°C potassium exposure. No significant weight changes are evident.

3. Conductor Continuous Performance Tests

Tests were completed on nickel-clad silver conductors in the two- and four-terminal test containers with the latter accumulating 917 hours of test time.

a. Two-Terminal Test Container

The two-terminal test container with the nickel-clad silver conductor (which was built and given tests previously*) was placed in a vacuum chamber which was then evacuated to a pressure of 2×10^{-6} torr. The container terminals were then energized at room temperature with a 2.5-volt, a-c, 90-ampere source. This voltage was impressed across the conductor and the increase in seal temperature was monitored. The potassium metal, which was deposited on the seals in previous tests, clinging to and covering the Lucalox insulation portion of these Westinghouse fabricated seals, continued to conduct. When the voltage was applied across the potassium bridging the terminal seals, the seal temperature increased very rapidly. Heating the seal by the passage of current from the seal terminal to the test container was not adequate to completely sever the potassium conductor. The maximum calculated seal terminal to container resistance, 25,000 ohms, was obtained after a measured voltage of 250 volts at a current of 10 milliamperes was impressed for three minutes. At this point, the potassium vapor ionized as evidenced by oscilloscope traces.

b. Four-Terminal Test Container

The four-terminal continuous conductor performance test container was assembled with rectangular nickel-clad

* See pages 19-25, Third Quarterly Technical Progress Report.[1]

FIGURE 1. As-Received Iridium Wire Showing Fibrous Structure After Breaking By Bending, 40X

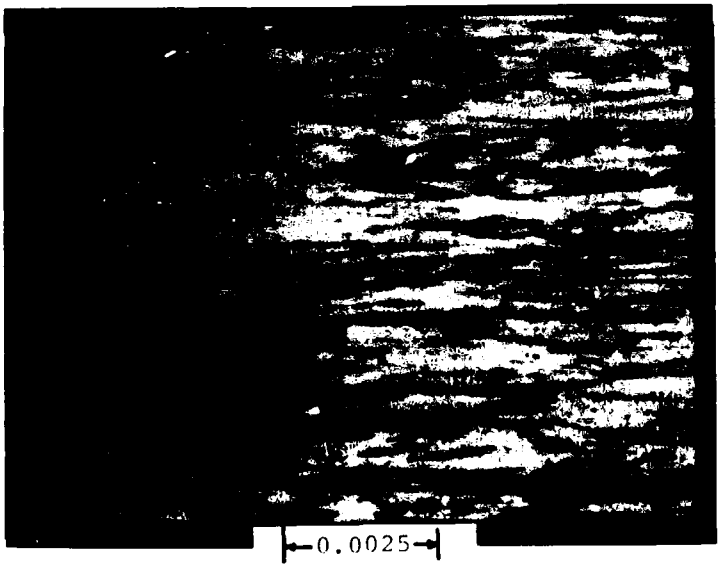


FIGURE 2. As-Received Iridium Wire, Longitudinal Section, 384X (Electrolytic Etch, H_2SO_4).

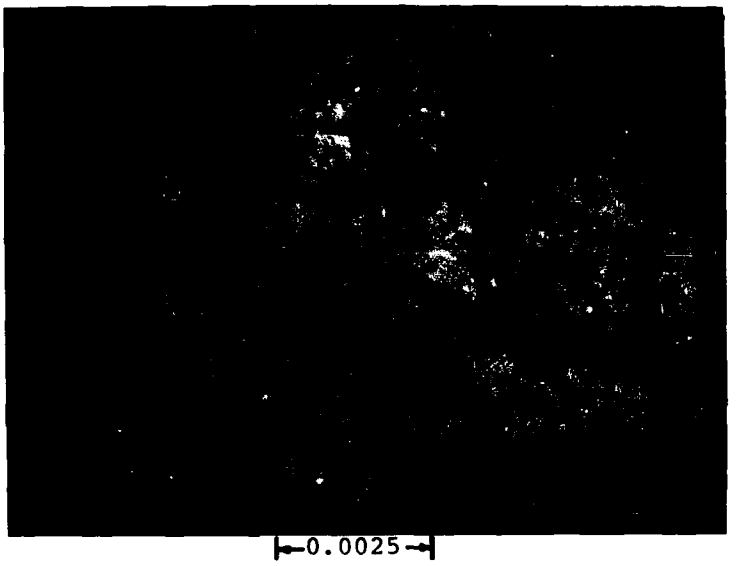


FIGURE 3. As-Received Iridium Wire, Transverse Section, 384X (Electrolytic Etch, H_2SO_4)

TABLE V. WEIGHTS OF METALLIC SPECIMENS BEFORE AND AFTER 1000-HOUR, 600°C POTASSIUM VAPOR EXPOSURE TESTS

Material*	Weight Before (gm)	Weight After (gm)	Weight Change (gm)	% Weight Change
Rhodium	3.5051	3.5063	gain 0.0012	0.034
Iridium	6.5547	6.5538	loss 0.0009	0.014
Nickel Clad Silver	47.8864	47.8864	-	-
Inconel Clad Silver	43.2380	43.2418	gain 0.0062	0.014
Cubex	10.5604	10.5604	None	-

* A more complete description of specimens is given in Table III.

silver conductor as the test specimen. The terminal seals used with this container were those obtained from the EIMAC division of Varian Associates.* Potassium was introduced into the test container through a fill tube. The test container was evacuated and sealed. Electrical connections were made to the four-terminal seals and to electrical feed through terminals located on the face plate of a hot wall vacuum furnace. The furnace was evacuated to 2×10^{-6} torr and resistance versus temperature readings were then obtained during heating. A plot of the resistivity measurements during this heat up are shown in figure 4. The temperatures of the test container, potassium vapor, and terminal seals were monitored during the test with thermocouples.

Electrical resistance as a function of the temperature had been previously obtained on the test conductor, in the container, in the absence of potassium vapor and is also shown on figure 4. Resistance values obtained in the absence of potassium were identical with values obtained on the initial temperature increase with potassium in the container.

* See pages 25 and 26 of Third Quarterly Technical Progress Report.[1]

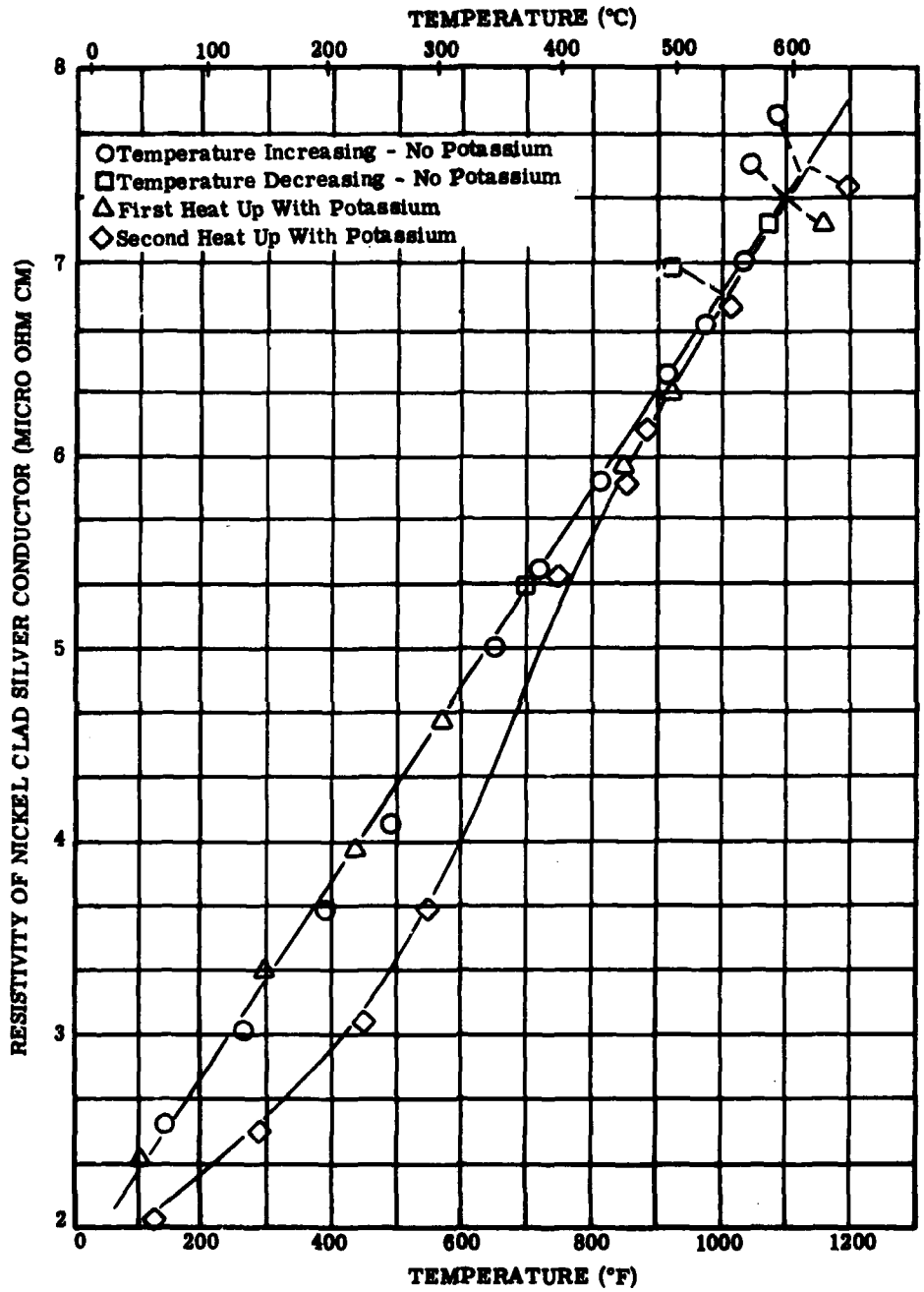


FIGURE 4. Apparent Resistivity of a Nickel Clad Silver Conductor as a Function of Temperature with and without Potassium in the Test Container

At a temperature of approximately 450°C, the potassium apparently wetted the beryllia insulator portion of the terminal seals (as it apparently had wetted the Lucalox insulator in the terminal seals of the two-terminal seal container) forming a conductor from seal terminals to ground. The conduction path at this temperature had a substantially higher resistance than the nickel-clad silver conductor and, therefore, did not appreciably affect the resistance of the test circuit as the temperature was increased to 607°C. After several hours at 607°C, the container was cooled to room temperature, removed from the furnace, and checked for possible potassium leaks. No leaks were found. The resistance from terminals to ground, measured with a Kelvin bridge, was 0.015 ohms. At this time terminal seal heaters were added to the test container.

The potassium loaded test container was then heated in the vacuum furnace a second time. The apparent conductor resistance readings varied greatly from the previous readings until a temperature approximately 450°C was reached. From 450°C to 600°C resistance readings were approximately the same as in the no potassium case. Figure 4 shows resistivity during this second heat up with potassium.

A seal heater was used to raise the temperature of the electrical terminal seals 150°C above the temperature of the potassium vapor. The resistance was measured from the terminal to container ground with the terminal seals at 750°C and the container at 600°C. The terminal seal to container resistance increased from 22 to 500 ohms; however, the contact was not broken.

A high impedance power supply was connected across the potassium conductive path from seal to ground.* The potassium was vaporized by this power supply and subsequently went to an ionized state. The seal temperature increased 71°C above the container temperature of 686°C when the vapor ionized. This ionized condition resulted in a voltage reduction across the plasma

* The high impedance supply was a series reactor current-limited supply incorporating a linear a-c reactor similar to that employed in mercury vapor lamp operation. The open circuit-short circuit characteristics of the supply were approximately 440 volts and 5.0 amps rms at 60 Hz.

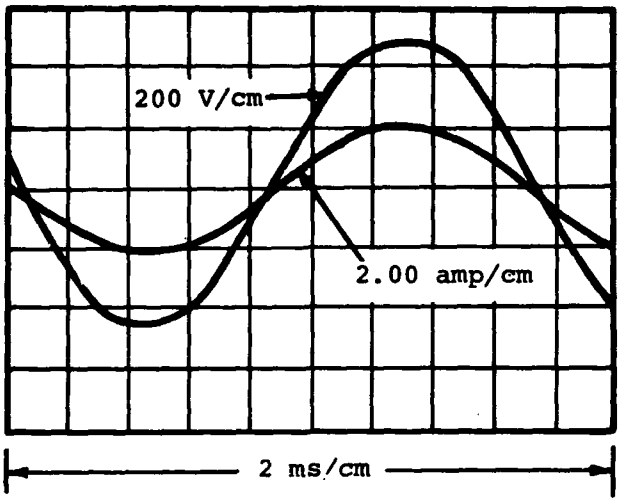
down to 27.5 volts rms, 60Hz and a current increase to approximately 2.5 amperes rms. Figure 5 shows the voltage and current before ionization. The seal temperature dropped as a result of the ionization because the input power was reduced. Figure 6 shows the voltage and current after ionization. (The corresponding temperature drop was from 757°C to 637°C.)

An effect was noted on the potassium plasma when the vacuum furnace heating coil was in energized and de-energized states. The magnetic field generated by the furnace was sufficient to cause the effective resistance of the plasma to change. The voltage across the plasma, at a constant current, increased from 27.5 volts a-c when the furnace was off to 50 volts a-c when the furnace was on, indicating an increase in effective electrical resistance. This could be explained by the magnetic field causing the plasma to spiral, increasing the mean length of the plasma, thus increasing its overall effective resistance.

The nickel-clad silver conductor was energized by 80 amps at 0.324 volts for 240 hours. There was no apparent degradation from this test.

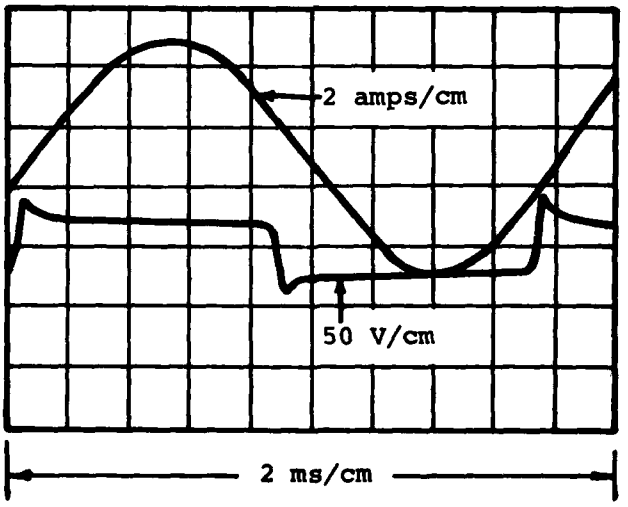
The potassium seemed to adhere to the beryllia walls of the terminal seals by some unknown mechanism. This effect appears to be a function of potassium concentration that is related to volume, vapor pressure, and temperature.

A total of 917 hours were accumulated while the four-terminal test container was at 600°C, or above. Resistance measurements were made on the test conductor, using the four-point method and a Kelvin bridge, at various times during the test. Resistivity values were calculated from these measurements and are shown in figure 7 with the temperature of the conductor at the time of measurement. An attempt was made to correct the resistivity of the conductor to a constant temperature of 600°C by applying a correction factor. This factor is the product of the difference between 1112°F and the test temperature times 5.1×10^{-3} ohm/°F (which is the slope of the upper portion of the curve in figure 4). The rectangles in figure 7 are the estimated resistivity at 1112°F for the nickel-clad silver conductor. A slight increase in resistivity is shown by the curve and in the order of 3.5% (based on resistivity at start and end of test) for the 917 hours.



Power = 440 watts

Figure 5. Traces of Voltage and Current Before Potassium Was Ionized



Power ≈ 27.5V x 2.5 amps

Figure 6. Traces of Voltage and Current After Potassium Was Ionized

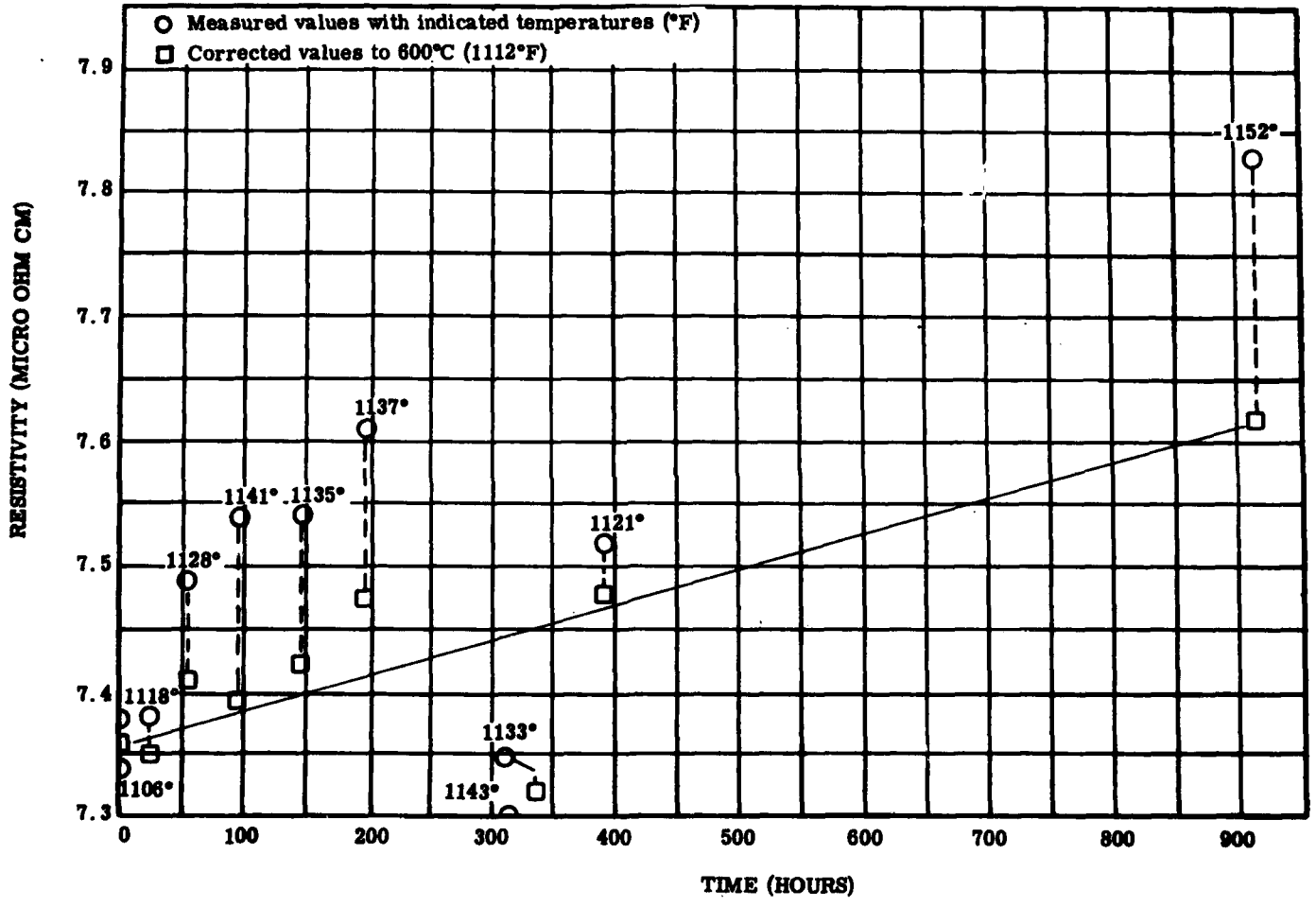


FIGURE 7. Resistivity as a Function of Time for Potassium Exposed Nickel Clad Silver Conductor

4. Rowland Ring Core Box Fabrication

A core box constructed of potassium resistant materials is necessary to contain Rowland rings for magnetic testing in the presence of potassium vapor. One element in the core box must be an electrical insulator to break the electrical circuit that would be created around the Rowland rings if the core box was constructed entirely of electrical conductor materials. Several core box designs using columbium-1% zirconium metal high alumina ceramic insulating materials are being evaluated.

a. Alite A-610 Washer Type Core Box

Additional vacuum furnace braze attempts were made using previously described Alite A-610 washers.* A different braze compound was used in the later brazes. This braze compound has the same nominal chemical composition as that used earlier for successful ceramic to metal joints**, but was compounded of unalloyed high purity powders. This braze gave good wetting of both metal and ceramic, but gave an unuseable product because of cracking of the Alite A-610 core box ceramic.

b. Lucalox Washer Type Core Box

A Lucalox washer was brazed to columbium-1% zirconium core box sides. This washer had a rectangular cross section and fit between core box sides. Around both the inside and outside core box sides were pieces of columbium-1% zirconium which were formed to hold the compounded braze alloy referenced above. The initial vacuum furnace braze attempt resulted in a leaking joint, possibly due to the braze cycle. A second braze attempt with a different braze cycle resulted in radial cracks across the Lucalox ceramic with braze metal filling the cracks thus forming a conductor between inside and outside core box sides.

c. Terminal Seal Type Core Box

The present approach to a Rowland ring core box design uses previously discussed terminal seal technology.***

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* Figure 23 page 37 of Second Quarterly Technical Progress Report.[3]

** Page 14 of Third Quarterly Technical Progress Report.[1]

*** See page 14 of Third Quarterly Technical Progress Report.[1]

Here, ceramic/metal brazed joints are in the materials and geometry of electrical feed through terminal seals with the core box ceramic element the inside cylindrical portion of the core box. Figure 8 shows the parts which will make up this core box. All metal parts are columbium-1% zirconium and the ceramic material is Lucalox. The braze joints were made with the unalloyed high purity powder combination. Assembly will be by TIG welding.

d. Bore-Seal Type Core Box

A backup core box design was created based on bore-seal type ceramic/metal materials and geometry. Figure 9 is a pictorial view of this type core box showing the core box ceramic as the inside cylindrical element.

B. PROCESS AND TECHNIQUES FOR POTASSIUM VAPOR RESISTANT ELECTRICAL DEVICE FABRICATION

1. General Description

Several materials and combinations of materials, applied in various ways, are being investigated as potassium vapor resistant electrical insulation for conductor and interlaminar applications. High alumina materials, aluminum oxychloride, and alimina-yttria eutectic are being either sprayed, radio frequency (R.F.) sputtered, or slip coated and fired on conductor or magnetic material substrates. The substrates include rhodium, iridium, and Hiperco 27 alloy. Nickel aluminide, zirconium, and titanium are being evaluated as a binder between the metal and the various ceramic insulations. Solid ceramics: alumina-yttria, yttria, strontium zirconate, gadolinia, samaria, and boron nitride are being evaluated for stability in potassium vapor.

Progress in the investigation of processes and techniques for fabrication of potassium resistant materials into electrical components is given in the following section.

2. Materials For Short-Term Potassium Vapor Exposure Test

Specimens of several materials were prepared for short term (200 hours) 850°C potassium vapor exposure testing. Some of these were loaded and sealed in potassium containing capsules.

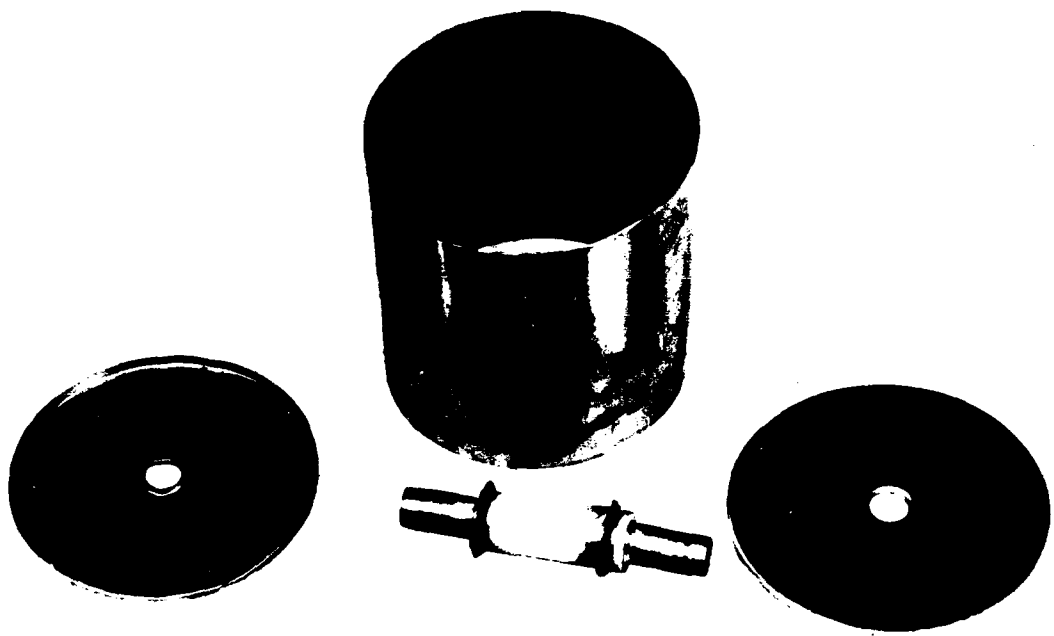

FIGURE 8. Terminal Seal Type Core Box Parts

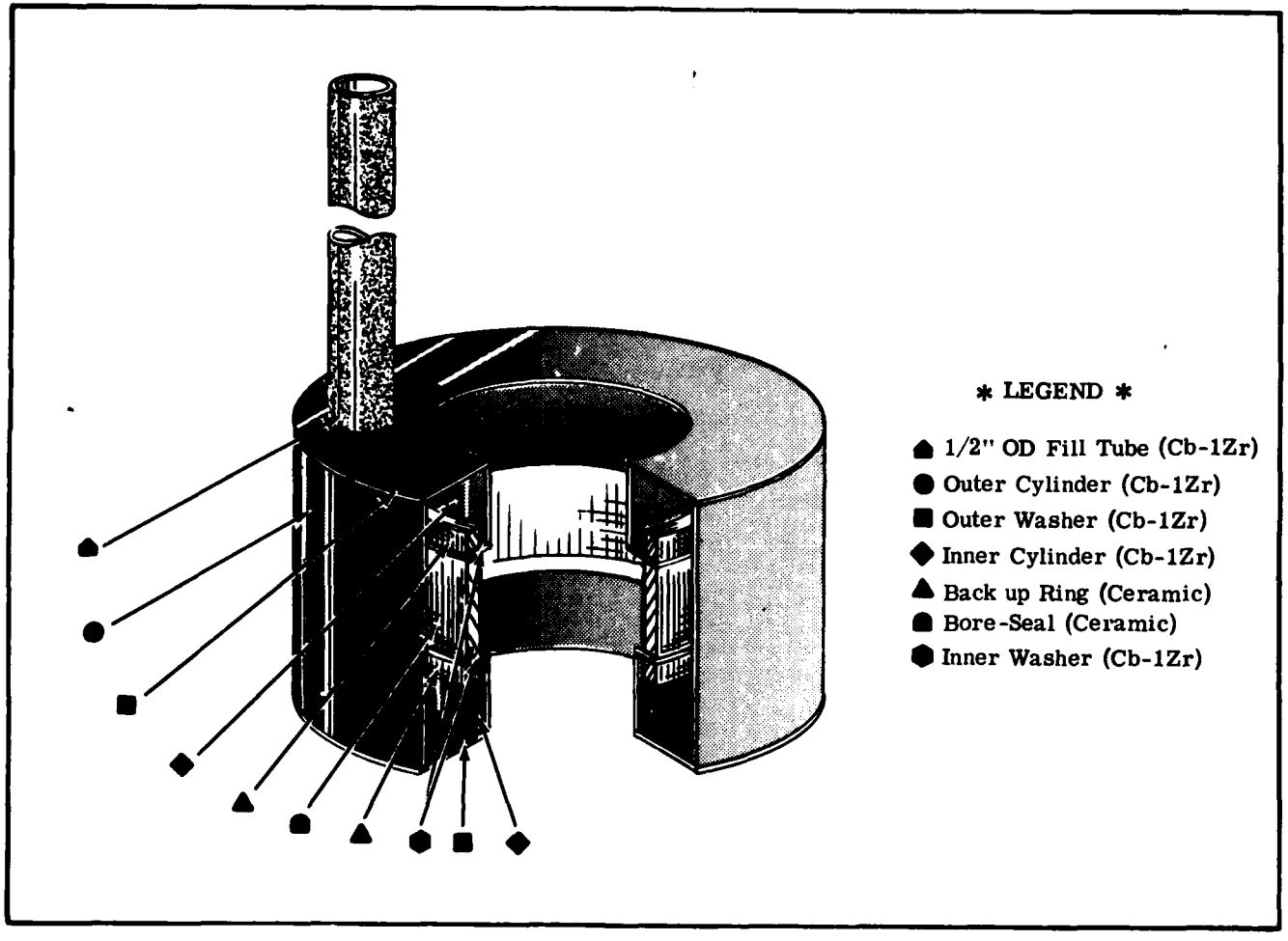

FIGURE 9. Bore-Seal Type Rowland Ring Core Box

Samarium oxide and gadalinium oxide modulus-of-rupture bars from previously prepared hot-pressed discs* were loaded into potassium containing stainless steel capsules. The following previously prepared ceramic/metal interface type specimens** were loaded into potassium containing stainless steel capsules:

a) Hiperco 27 alloy substrates each with a sputtered interface layer of high purity titanium followed with a sputtered layer of Lucalox.

b) A substrate as above with the addition of a high purity alumina coating plasma arc sprayed in air.

The following specimens were prepared and are ready for loading into potassium containing capsules:

c) Substrates as a) above but vacuum heat treated at 1800°F for 30 minutes.

d) Substrate as b) above but vacuum heat treated at 1800°F for 30 minutes.

Specimens from a) and c) above will be evaluated for interlaminar insulation properties and b) and d) to determine the protection afforded the Hiperco 27 alloy substrate by the sputtered coatings when plasma arc sprayed in air. Specimens from c) and d) will be compared in the a) and b) to determine the effectiveness of the heat treatment.

Boron nitride modulus-of-rupture bars were prepared from Raytheon CVD (chemically vapor deposited) high purity material. These bars were cleaned and weighed and stored in a desiccator prior to loading in potassium vapor exposure test capsules.

3. Ceramic/Metal Interface Development

The application of potassium vapor resistant interlaminar and conductor insulations by processes other than plasma arc spraying and sputtering were investigated. These processes were: bonding alumina by chemical reaction, depositing alumina by chemical decomposition, and bonding a eutectic mixture by melting it.

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* See page 34 - Third Quarterly Technical Progress Report.[1]

** See pages 32-34. Third Quarterly Technical Progress Report.[1]

a. Alumina Interlaminar Insulation

An interlaminar insulation investigation was conducted based on binding high purity alumina to Hiperco 27 alloy substrates by reaction with titanium from titanium hydride. The substrates for this investigation were Hiperco 27 alloy transformer E laminations either with or without nickel plating. These substrates were chemically cleaned with acetone prior to the application of the titanium hydride. The titanium hydride was in the form of -2 micron particle size powder. This material was mixed with a 10% amyl acetate-methyl cellulose solution. Both paint and spray application techniques were investigated. Spraying with an air brush gave the most uniform coating.

High purity alumina was used in the forms of 0.05 micron size (Linde B) and 0.3 micron size (Linde A), both in 10% amyl acetate-methyl cellulose binders. Little difference could be detected in handling the two particle size powders. Brush application tended to remove the layer of titanium hydride thus air brush spraying was used to prepare specimens. Both the titanium hydride and alumina coatings were air dried at 200°F for one hour before further processing.

Specimens both with and without the nickel plating were given one of the following heat treatments.

1) 1000°C for 10 minutes in a hydrogen atmosphere furnace, cooled in nitrogen.

2) 1000°C for 10 minutes in a nitrogen atmosphere furnace, cooled in nitrogen.

3) 1000°C for 10 minutes in evacuated retort, cooled in vacuum.

In all cases the alumina failed to adhere to the substrates. A gray to black powder resulted with the lighter color being on the nickel plated substrates. The probable cause of the lack of adhesion is the incomplete decomposition of acetate-methyl cellulose binder.

b. Alumina Conductor Insulation by Chemcial Decomposition

Previously prepared aluminum oxychloride* was applied to 0.030-inch diameter rhodium conductors by flowing

* See pages 37-41. Third Quarterly Technical Progress Report.[1]

on a slurry of aluminum oxychloride in absolute ethyl alcohol and air drying. Wires were resistance heated in air by high current, low voltage alternating current. Wire temperature was monitored by an optical pyrometer. The rhodium wire was heated to temperatures as high as that required to melt rhodium, 1960°C.[4] During heating the aluminum oxychloride coating underwent decomposition giving a densified alumina coating which appears to have a sintered structure. The coating is not well bonded to the rhodium and can be removed by scraping. Figure 10 shows rhodium wire with an alumina coating from the decomposition of aluminum oxychloride.

c. Alumina-Yttria Conductor Insulation

Both pre-fused and mixed-calcined powders of alumina-yttria in the ratio of 82 mole % Al_2O_3 - 18 mole % Y_2O_3 were applied to rhodium conductor material. The pre-fused material was the vacuum melted disc reported earlier* which was crushed in a diamond mortar and milled using a Pitchford Blender Mill with steel balls. The resulting average particle size was five microns. Both the crushing and the milling contaminated the material with steel. Steel was removed by boiling the powder in a 50% mixture of hydrochloric acid and distilled water and an attempt was made to filter out the powder. The powder went through the filter. Separation was made by rinsing and settling the powder and decanting off the liquid. After several rinses the powder was made into a slurry with absolute ethyl alcohol. The mixed-calcined powder was from the same material used in making hot-pressed discs.** This material was used in a slurry with distilled water.

Both the pre-fused and mixed-calcined powder were applied to rhodium wire from slurries. Both brushing and dipping were used to apply the slurries with dipping giving the most uniform coating. Three coatings with an air dry between each coating were used to prepare the wires. Heating was accomplished in air by passing a high alternating current at a low voltage through the wire. At a temperature near the melting temperature of rhodium the pre-fused material melted and was drawn into droplets which adheared to the surface of the oxidized rhodium. Figure 11 shows the surface of

* Pages 23 and 24 First Quarterly Technical Progress Report.[3]

** Page 34 Third Quarterly Technical Progress Report.[1]

FIGURE 10. Alumina From Aluminum Oxychloride on Rhodium Wire (40X)

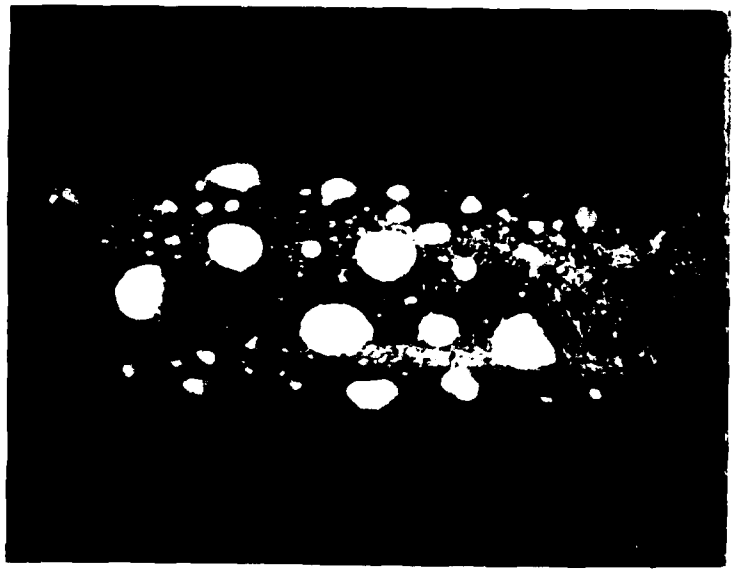


FIGURE 11. Alumina-Yttria from Pre-fused Material on Rhodium Wire (40X)

the oxidized rhodium and fused droplets of alumina-yttria near the point where the rhodium wire melted. The mixed-calcined powder did not fuse or adhear when heated to the melting temperature of the rhodium wire.

4. Properties of Hot-Pressed Materials

Hot-pressed ceramic materials which were made at Westinghouse were given density tests and microstructure examinations.

a. Density Measurements

Density measurements were made on hot-pressed materials prepared for short-term potassium vapor exposure tests. Specimens were weighed in air and liquid (carbon titrachloride) and a correction was made for the weight of the platinum wire (specimen holder) suspended in the liquid. Densities were calculated from the following relationship:

$$\text{Density } (\text{gm/cm}^3) = \frac{\text{weight in air}}{(\text{weight in air}) - (\text{weight in liquid})/(\text{density of liquid})}$$

These data are shown in table VI. Calculated densities are compared to theoritical values except for the alumina/yttria material. The accuracy of the measuring technique was checked by running several samples of Linde sapphire. Calculated densities were 3.98 and 3.96 gm/cm^3 compared to a theoretical value of 3.98 ± 0.02 for hexagonal alumina.

b. Microstructure

Photomicrographs were made of a number of different hot-pressed materials primarily to compare grain size and to indicate significant structural differences. Figures 12 to 16 show the microstructures of Y_2O_3, chemically pure (CP) $SrZrO_3$, pre-fused $SrZrO_3$ and Al_2O_3-Y_2O_3 respectively at magnifications of 1600x or 1000x.

Listed below are the estimated grain sizes of these materials:

1) Y_2O_3 - 2 to 3 microns

2) Sm_2O_3 - 16 to 19 microns

3) CP $SrZrO_3$ - 7 to 9 microns

TABLE VI. DENSITY MEASUREMENTS OF HOT-PRESSED CERAMIC SPECIMENS

Material (Process Number)	Sample Weight		Calculated Density (gm/cm^3)	Theoretical Density[2] (gm/cm^3)	Percent of Theoretical (%)
	Air (grams)	Liquid[1] (grams)			
Y_2O_3 (K1935131)	0.4831	0.3305	5.02	5.03 (Cubic)	99.8
Gd_2O_3 (K 1935133)	1.7238	1.3613	7.52	7.63 ± 0.01 (Cubic)	98.6
Sm_2O_3 (K 1935132)	2.5512	2.0215	7.62	7.62 (Cubic)	99.9
$SrZrO_3$ - From Chemically Pure Powder (K 1935128)	1.4684	1.0381	5.43	5.48 (Cubic)	99.2
$SrZrO_3$ - From Pre-Fused Powder (K 1935128)	1.3779	0.9621	5.26	5.48 (Cubic)	95.7
Al_2O_3 / Y_2O_3 K 19135135)	1.3277	0.8256	4.23	Unknown	--
Al_2O_3 (Linde Sapphire)	0.5791	0.3487	3.98	3.98 ± 0.02 (Hexagonal)	100
Al_2O_3 (Linde Sapphire)	3.5574	2.1382	3.96	3.98 ± 0.02 (Hexagonal)	100

(1) Carbon Tetrachloride, Fisher Reagent, Density (gm/cm^3) 1.584 ± .001 @ 25°C

(2) J. F. Lynch et al (ED.), Engineering Properties of Selected Ceramic Materials, Journal of the American Ceramic Society, 1966.

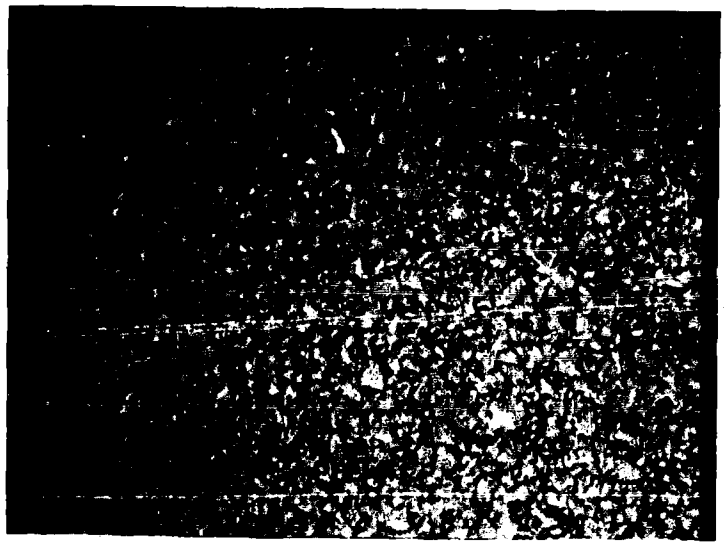


FIGURE 12. Microstructure of Hot-Pressed Yttria (1600X)

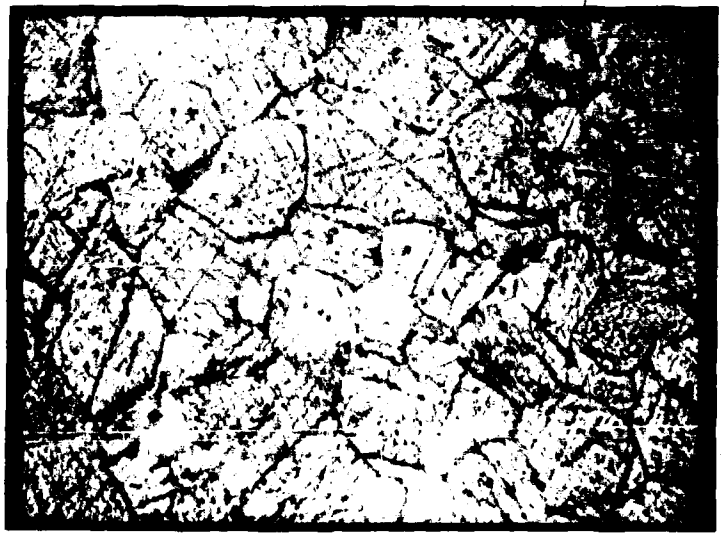
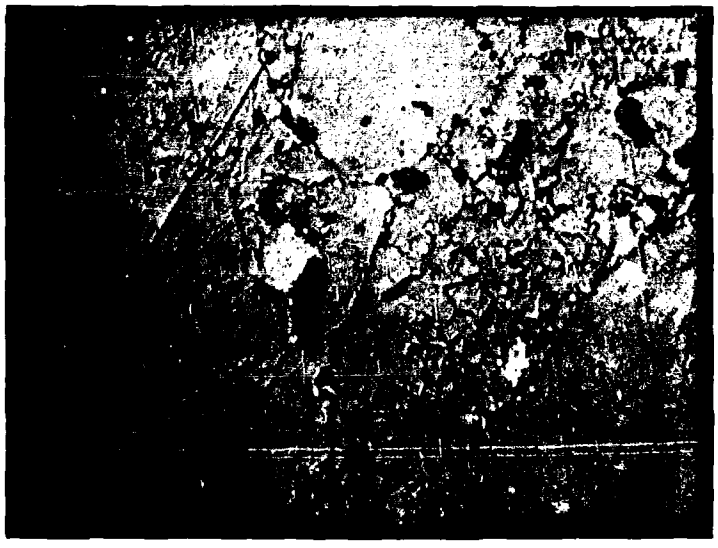


FIGURE 13. Microstructure of Hot-Pressed Samaria (1000X)

25μ

FIGURE 14. Microstructure of Hot-Pressed Chemically Pure Strontium Zirconate (1600X)



25μ

FIGURE 15. Microstructure of Hot-Pressed Strontium Zirconate (from Pre-fused Powder) (1000X)

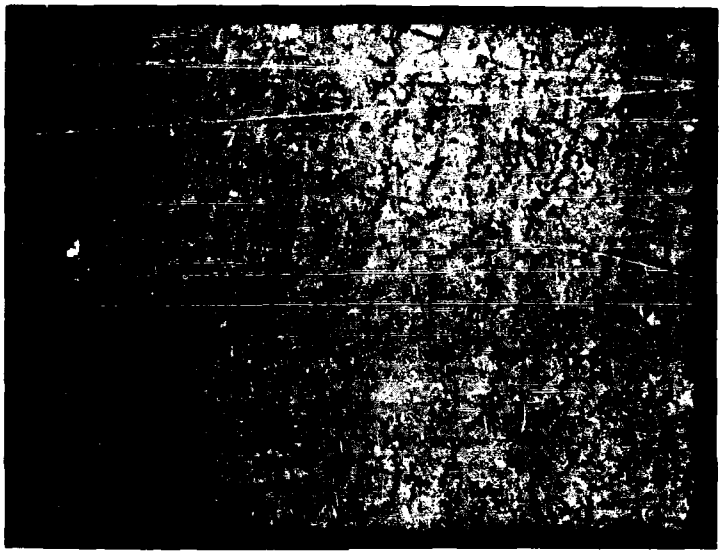


FIGURE 16. Microstructure of Hot-Pressed Alumina-Yttria (1000X)

4) Pre-Fused $SrZrO_3$ - 6 to 25 microns

5) Al_2O_3-Y_2O_3 - 2 phases

All materials have a crystalline structure except the Al_2O_3-Y_2O_3 eutectic which appears to be a two-phase system. One phase has a high population of residual polishing scratches (see figure 16) indicating that it is relatively softer than the other phase.

C. FABRICATION AND TESTING OF FIVE-KVA TRANSFORMERS

1. General Description

Two five-KVA transformers will be built using Hiperco 27 alloy magnetic material, Linde 99.9% Al_2O_3 interlaminar insulation, rectangular nickel-clad silver conductors, and an alumina conductor insulation. The transformers will be evaluated in potassium vapor at 600°C; one of these in ionized potassium vapor. The life objective is 500 hours operation for the un-ionized vapor test.

Progress in fabrication and evaluation of transformers is given below.

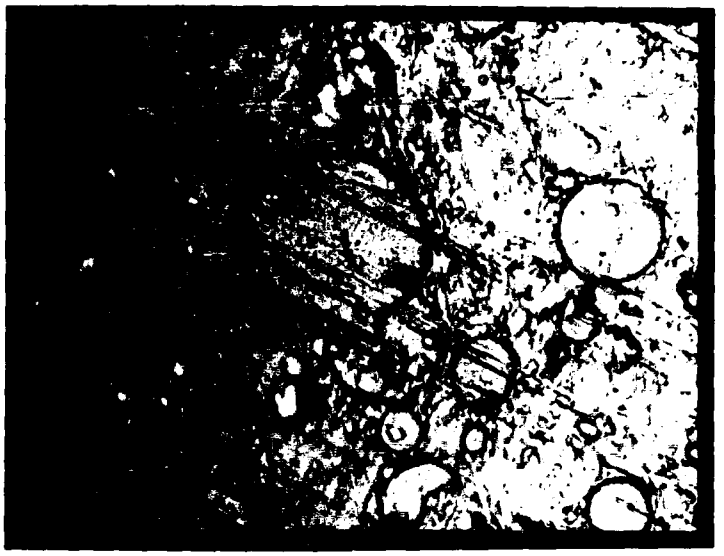
2. Transformer Magnetic Material

A program was undertaken to develop the parameters necessary to obtain a suitable magnetic material interlaminar insulation by plasma arc spraying high purity alumina in an inert atmosphere chamber. Problems were encountered in maintaining a high purity argon atmosphere, getting suitable coverage, suitable adhesion, and suitable insulation properties. Figure 17 shows one of the best coatings developed to date with bright and dark field illuminations. This coating could not be duplicated due to copper particles from the plasma arc spray gun which were deposited with the alumina on subsequent runs.

3. Transformer Electrical Conductors

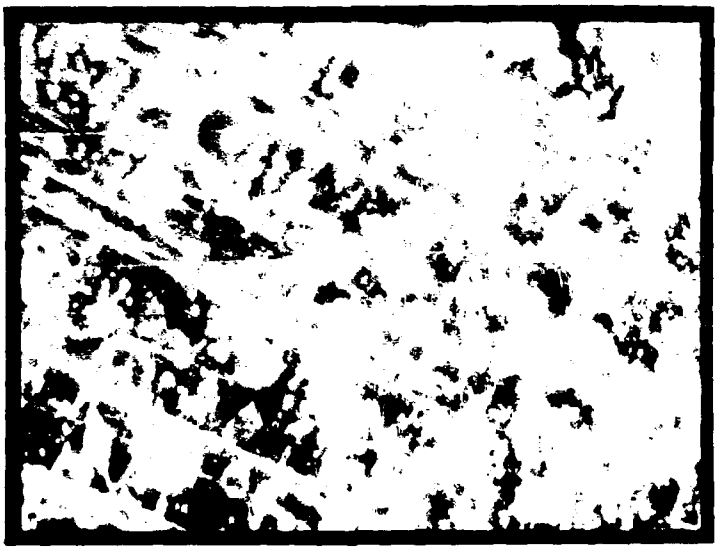
A fixture for winding transformer conductors was designed and fabricated. One layer of nickel-clad silver conductor consisting of two concentric windings were prepared on this fixture. These windings were vacuum annealed at 800°C for 30 minutes to reduce spring-back.

Additional nickel-clad silver conductor material was received. This is new material, i.e. not squared circular conductor material from the previous program.



⊢0.00125⊣

Bright Field Illumination



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Dark Field Illumination

FIGURE 17. Plasma Arc Sprayed Spherical Alumina on Hiperco 27 Alloy Substrate (800X)

WAED 67.29E-35

4. Sapphire Window Seals

Attempts were made to braze tapered sapphire windows to flared columbium-1% zirconium tubes. Only the unalloyed braze alloy was used. Braze attempts were made in a cold wall radiant heated furnace pumped by a sputter ion pump and in a RF induction heated cold wall furnace pumped by an oil diffusion pump. Various heating cycles were used with each furnace. Good wetting of both materials by the braze alloy were accomplished in the radiant heated furnace, but a leak tight joint was not obtained. Good wetting of both materials was obtained in the RF induction heated furnace but a good seal was not obtained due either to leaks or broken sapphire windows. A certain percentage of sapphire windows, as-received from the supplier, apparently contain flaws that cannot be successfully detected until active metal brazing shows the flaw as a crack after the brazing cycle.

SECTION IV

PROGRAM FOR NEXT QUARTER

A. EVALUATION OF POTASSIUM EXPOSED MATERIALS

1. Long Term Tests

Long term potassium vapor exposure tests will be continued with all tests completed except the 5000-hour test. The 1000, 2000, and 3000-hour test specimens will be evaluated. A chemical analysis of ceramic materials will be performed.

2. Rowland Ring Tests

Rowland rings will be given plasma arc sprayed high purity alumina interlaminar insulation. A Rowland ring core box will be assembled. Rowland rings will be given magnetic test at 600°C in vacuum.

3. Conductor Continuous Performance Tests

The two-terminal and four-terminal test containers will be opened, potassium neutralized and removed, and the conductors removed and examined. Terminal seals will also be examined.

B. PROCESSES AND TECHNIQUES FOR POTASSIUM VAPOR RESISTANT ELECTRICAL DEVICES

1. Ceramic/Metal Interface Development

Additional ceramic/metal interface specimens will be prepared on Hiperco 27 alloy magnetic material and on conductors. Pre-fused milled alumina-yttria will be coated on rhodium conductor and processed in a vacuum furnace and by resistance heating in air. Aluminum oxychloride will be coated on nickel clad silver and rhodium conductors, and on Hiperco 27 alloy.

2. Short Term Potassium Vapor Exposure Tests

Short term 850°C potassium vapor exposure tests will be conducted on those ceramic and ceramic/metal interface specimens which have been prepared for test.

C. FABRICATION AND TESTING OF THE FIVE-KVA TRANSFORMERS

1. Transformer Magnetic Material

Parameters will be established for plasma arc spraying suitable high purity alumina interlaminar insulation coating in a recirculating inert atmosphere chamber. Transformer E and I laminations will be given interlaminar insulation.

2. Transformer Electrical Insulation

Transformer Alite A-610 electrical insulations will be analyzed and their suitabiility for the intended purpose determined.

3. Transformer Electrical Conductors

New nickel-clad silver conductor material will be annealed. Work will be continued on winding and insulating transformer electrical conductors of nickel-clad silver.

4. Sapphire Window Seals

Additional attempts will be made to fabricate helium leak tight sapphire windows.

SECTION V

REFERENCES

1. Third Quarterly Technical Progress Report, Alkali Metal Resistant Electrical Devices, WAED 67.13E

2. First Quarterly Technical Progress Report, Alkali Metal Resistant Electrical Devices, WAED 66.46E

3. Second Quarterly Technical Progress Report, Alkali Metal Resistant Electrical Devices, WAED 66.62E

4. Englehard Industries, Inc., Technical Bulletin, Volume VI, No. 3, December 1965.